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PATENT SPECIFICATION

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806,362

Date of Application and filing Complete Specification: Nov. 7, 1956.

No. 34055/56.

Complete Specification Published: Dec. 23, 1958.

Index at acceptance:—Class 70, E6A.

International Classification:—B29j.

COMPLETE SPECIFICATION

An improved process for Incorporating Carbon Black and other Compounding Ingredients in Rubber

We, MARCO COMPANY, INC., a corporation organized and existing under the laws of the Commonwealth of Pennsylvania, of 1000 Hess Street, Saginaw, County of Saginaw, State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to an improved process for incorporating carbon black and other compounding ingredients in rubber latex.

An object of the invention is to compound rubber when in a liquid suspension and avoid the conventional compounding step of mastication of rubber and compounding ingredients, and then to separate the compounded rubber from the liquid medium.

A further purpose is to reduce the power requirements for machinery conventionally used to disperse carbon black and other compounding ingredients in rubber.

A further purpose is to avoid the harmful effects of oxidation or degradation during the process of incorporating carbon black or other compounding ingredients in dry rubber polymer.

A further purpose is to obtain more uniform dispersion of the added components in the polymer.

A further purpose is to reduce the space requirements for rubber compounding as compared to conventional equipment.

A further purpose is to afford the operator an opportunity to select through instrumentation component relationships needed for particular rubber compounding recipes.

The colloidal rubber dispersions which are prepared according to the present invention insure against possible agglomerates or aggregations in the end product.

The rubber compounding process of the present invention is divided into several phases in order to obtain greater homogeneity of the end product.

The process of the present invention permits the use of various types of latexes to suit the requirements of the desired end product.

The present invention provides for coagulating continuously the pre-mixed components in order to control the particle size.

If an oil (extended) is to be incorporated into the rubber latex, the oil, according to the present invention, can be incorporated as such into the rubber latex by means of metering the oil directly into the latex and homogenizing the mixture without necessitating prior emulsification of the oil in water or caustic solution.

The drawings illustrate mechanism which may be used in carrying out the process.

Figure 1, is a diagrammatic flow chart of mechanism which may be employed in carrying out the process of the invention.

Figure 2 is a partially diagrammatic axial section of a homogenizer and secondary mixer useful in the process of the invention.

Figure 3 is a section on the line 3—3 of Figure 2.

Describing in illustration but not in limitation and referring to the drawings:

In current rubber compounding practice, carbon black and other compounding ingredients including other pigments are usually incorporated into the rubber by plastic mastication in a mechanism such as a Banbury or Sigma-blade mixer and finally completing the operation on a finishing mill.

The equipment required for this procedure is elaborate and costly, requiring heavy foundations and occupying considerable room. Specially competent labor is required to operate the equipment and the conditions of operation are unpleasant due to the large amount of dust produced. Considerable labor is required for weighing out components which are to be used in the compounding recipe. The power requirements are also high and vibration is a serious problem.

Efforts in the past have been made to incorporate pigments with rubber latex in the presence of water, and then masticating the final product after removal of the water. To

this, usually, other additives are incorporated, necessitating a second operation and not obtaining the same comparative homogeneity as our process.

In the prior art, where oil (extender) is incorporated, it is usually first mixed with resin, soap, fatty acid, etc., and then emulsified in dilute caustic soda solution. The emulsion is then incorporated into the rubber and mixed by mastication.

According to the present invention, the entire compounding operation is carried out continuously in liquid phase, and plastic mastication in a Banbury mixer or otherwise is avoided.

Each of the compounding ingredients other than the carbon black (except as noted below) is separately milled to colloidal particle size with liquid medium (such as water) to obtain a separate colloidal dispersion of the compounding ingredient in that medium. Then the resulting slurries are separately metered to a common point. Carbon black, and liquid medium therefor, are also metered to this common point. In one embodiment of the invention, the carbon black is introduced as measured dry powder and its liquid medium is introduced separately. In another embodiment of the invention the carbon black is first milled to colloidal particle size in its liquid medium to form a colloidal dispersion and this colloidal dispersion is metered to the common point.

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A10

Oil (extender) will desirably be metered as liquid oil to the common point, and there mixed with the liquid ingredients. The short time of the entire operation aids in maintaining the oil in droplet form emulsified in the liquid medium without need for emulsifying agents.

At the common point a master dispersion mixture of the compounding ingredients other than carbon black is formed.

It will of course be evident that the compounding ingredients other than carbon black may be other pigments, or fillers such as clay, reclaimed rubber, etc., or may be reacting ingredients such as sulphur, zinc oxide, or the like. It will also be evident that the individual slurries of the compounding ingredients when prepared may be stored in suitable storage tanks until they are to be introduced into the master dispersion mixture.

Since the other compounding ingredients have formed separate colloidal dispersions, prior to their mixing together, the resulting mixture is relatively free from aggregates or agglomerates.

If the carbon black has been added dry, the master dispersion mixture is first incorporated and then intensively mixed, while if the carbon black has been added as a colloidal dispersion the master dispersion mixture is first homogenized and then intensively mixed.

After the mixing of the master mixture, the master batch mixture is milled to colloidal particle size so as to form a master dispersion. The master dispersion and latex are then separately metered to another common point and the master dispersion and the latex are then homogeneously mixed so as to impregnate the particles of compounding ingredients into the rubber latex.

All of the above operations beginning with the combining of the ingredients into the master dispersion mixture through to the final homogeneous mixing are accomplished continuously and without any interruption. The purpose of the final homogenizing is to eliminate any possible clumping and thus avoid necessity for plastic masticating in a Banbury or otherwise.

An important aspect of the invention is that from the time that the blending of the ingredients into the master dispersion mixture takes place, there is no interruption or quiescent period, as this would be an opportunity to form pockets with short circuiting, so that the components would not distribute uniformly but become segregated.

The final homogenized liquid phase compounded rubber latex is then desirably carried through a so-called reactor including stators and impellers, which includes a metering pump for adding coagulating agent in a strictly assured uniform association with the components. In this way the size of the crumbs or coagulated particles can be controlled, the velocity of the coagulating agent and the speed of the impellers as well as the concentration of coagulating agent being factors. The crumbs can be as small as 100 mesh or they can be as large as ½ inch in diameter.

In the preferred embodiment of the invention, temperature regulators supplying heating or cooling medium in suitable jackets or the like are employed on the various apparatus units. Usually, cooling is employed as for example to eliminate the possibility of encouraging acceleration particularly when ingredients such as zinc oxide and sulphur are present.

Whereas the mixing procedure involving dry mastication in the prior art develops very considerable heat, there is much less difficulty through temperature rise encountered in the present invention, and in fact the temperature can be held at any desired low level.

It will be evident from the above brief outline that the process of incorporating carbon black and other like particles of pigments into rubber has been greatly simplified, facilitated and reduced in cost.

Since the process of the invention is continuous rather than intermittent, much better control is obtained. This is particularly true of control of temperature. The mixing in liquid form itself tends to reduce the danger of developing excessive heat. Since the process of the present invention is carried out in

completely enclosed chambers, destructive oxidation during dispersion of the carbon black is not encountered.

The equipment required for mixing in the present invention is much simpler, smaller and more compact than the prior art equipment used. No heavy foundations are required and no substantial vibration is produced. Horsepower requirements for operation are lowered.

Rubber latex used in the present invention may be natural rubber latex or synthetic rubber latex such as GRS latex or neoprene latex or combinations thereof.

The carbon black may be any one of the carbon blacks used in rubber compounding from the very loose pellet type to the extremely finely divided type.

In accordance with the present invention, the particles of carbon black will be separate and completely surrounded by liquid.

The oil extender will suitably be a mineral oil such as Circosol. From 5 to 35 percent by weight on the rubber solids will commonly be used, preferably about 15 percent.

The end product may be a coagulated, pressed or crumbled product, and crumbling and coagulation will very desirably be carried on continuously as later explained more in detail.

The liquid medium referred to herein will usually be water. When oily or like materials are to be incorporated in the rubber latex, however, the liquid medium may then be, in some cases, an emulsion, for example, of petroleum or fatty oil in water. Dust and oil extender may be incorporated in the water in the form of a dispersion.

Referring to Figure 1, each of the compounding ingredients other than carbon black, such as clay, zinc oxide, sulphur, and the like is metered by a dry feeder into a mixer, along with liquid medium such as water, metered by a rotary pump. The ingredients are mixed in the rotary mixer and are then milled to colloidal particle size in a colloid mill. The ingredients thus formed into colloidal slurries are separately stored in tanks or the like.

From the storage tanks (not shown), the slurries are drawn by pipes 21, 22 and 23 to metering pumps 24, 25 and 26. Any suitable type of metering pump may be used.

From the respective metering pumps the slurries of colloidal compounding ingredients in the liquid medium pass respectively by pipes 27, 28 and 30 to a hopper 31 of a rotary mixer 32.

The mixer 32 is desirably of the character provided with a reactor head by which the mixture under pressure is forced through openings as shown in Figure 3 of the drawings at 70. The mixer or homogenizer 32 is in a sense a pump with a restricted outlet, as it contains a valve under spring tension placed within a wall which forms an anvil against which a stream of particles in the medium can impinge as the valve is released from the seat.

The mixer accomplishes a continuous mixing and turning and particularly a blending action on the material to be mixed.

Carbon black suitably in the form of an impalpable powder is placed in hopper 33 and fed to hopper 34 from which it is discharged by metering screw conveyor 35 at the bottom of hopper 34, operated by motor 36 and speed reducer 37. The screw conveyor discharges into hopper 31 where the slurries also enter. The screw conveyor performs a dual function. It not only meters the carbon black but it compresses the powder, eliminating contained air which would be objectionable, and forming from the powder a seal against splashing of slurry into the dry feed hopper 34, which would tend to clump the dry powder, encourage formation of gas pockets, and tend to produce dry core aggregates which would be difficult to break up.

The carbon black and the combined slurries enter the mixer 32 simultaneously and undergo churning, blending and other continuous mixing, the resulting mixture then being expelled under pressure through the outlet orifice in head 38 of the intensive mixer section. From this the master dispersion mixture then passes by pipe 40 through colloid mill 41, which contains shear plates and acts to break down or subdivide particles suspended in the liquid medium depending upon the running clearance and the rotor speed. The film going through the colloid mill is usually of the order of one or a few tenths of thousandths of an inch in thickness. The treatment in the colloid mill is characterized by shearing suitably accompanied by grinding and disintegrating causing a thorough dispersion of the carbon black in the liquid medium so that all particles are thoroughly wetted by the liquid medium due to the hydraulic and mechanical forces applied. Grinding action takes place between a rotor plate and two stator plates with discharge at the periphery.

The master colloidal dispersion which comprises carbon black and the other compounding ingredients in the mixture of liquid media leaves colloid mill 41 by pipe 42 to enter continuous mixer 43 which may be generally of the character of continuous mixer 32 above described. Pipe 42 enters hopper 44. Latex from pipe 45 connected with a suitable tank is metered by metering pump 46 which may be of the character of metering pumps 24, 25 and 26, and fed through pipe 47 to inlet hopper 44 of the continuous mixer 43. The slurry of the compounding ingredients and the rubber latex undergo churning, blending and general mixing action in the continuous mixer 43 and then are expelled through head 48 which may suitably be of the character of head 38 and including a valve acting against a seat under pressure and displaced to allow a thin

annular film flow to impinge against an anvil under pressure. This serves to partially homogenize the slurry.

The effluent from head 48 passes through pipe 50 to a homogenizer 51, which consists of a plate with an inlet opening, through which the material is fed to a rotor, where it is carried around under pressure, then forced against the tension of a spring-held valve and discharged under pressure between the valve element and seat, and impinges against an anvil.

The mixture of latex and the compounding ingredients, homogeneously associated, leaves the homogenizer by pipe 52 and suitably may go to any other equipment, the process thus far forming a complete compounding operation without requirement of solid plastic mastication.

It will be evident that from the time that the individual slurries have been brought together with the carbon black, the process goes on continuously and uninterruptedly and this is a major factor in avoiding difficulties through segregation and formation of dry core aggregates. The process from initial mixing at the common point to coagulating normally requires less than 10 minutes. Testing can be carried out and check backs and modifications in proportion can be made reliably for the purposes of control.

In the preferred embodiment, a coagulating agent, suitably dilute sulphuric acid, is introduced from inlet pipe 53 through coagulating agent pump 54 to coagulating reactor 55 into which pipe 52 also is introduced. Crumbs of rubber ready for filtering, screening or centrifuging pass out through pipe 56. The crumbs can be washed on a screen or on a filter and then run through a drier before moulding.

A suitable construction for reactor 55 is shown in Figures 2 and 3.

In this form a conical housing 57 is provided with a conical screw 58 which turns under suitable driving action of a motor (not shown) driving the screw shaft 60. The shaft is mounted by means of journal bearing 61 and thrust bearing 62 and is sealed by packing 63 compressed by spring 64. From the space inside of housing 57 the material being mixed passes through clearance 65 into an annular space 66 within a housing 67 having a series of axially spaced stator plates 68 provided with transfer holes 70, and intermediate rotors 71 having spoke-like blades radial to the axis of rotation. A packing is provided at 73 compressed by spring 74 and an outboard journal bearing is provided at 75 and a thrust bearing at 76. Inlet for sulphuric acid is provided at 77 and outlet for the final product at 78. The housing 57 is jacketed for temperature control at 80 and the housing 67 has temperature control piping connections at 81.

It will be evident from the above discussion that the mixing and compounding of the ingredients into the rubber is accomplished in liquid phase completely from beginning to end. There is no tendency to form dry core agglomerates which would be difficult to break down.

Ingredients are not merely agitated together but they are milled to colloidal size separately and then milled to colloidal size together. This is a further protection against forming dry core aggregates.

The separate milling of the ingredients with the water or other liquid media is very advantageous. If ingredients of different characters were initially colloidally milled together there would be a tendency to segregate, which is avoided in the present process.

Each of the ingredients is metered as the slurry is formed and also as the master dispersion mixture is formed. Likewise the carbon black is metered either in dry form or as a slurry.

The milling of the master dispersion mixture as such is very important, as this eliminates the least tendency toward stratification and eliminates clumping.

The resultant product is so excellent that the rubber granules after compounding and coagulating can be rubbed over a piece of white cotton fabric without forming any black smudge adherent to same.

It will be evident that the particles of the compounding ingredients are actually impregnated into the rubber latex by the mixing and colloid milling procedure above described.

The following is an example of one procedure in accordance with the present invention:

9% by weight of carbon black is dispersed in 91% by weight of water in a preliminary mixer and colloid mill combination. 1,319.5 lbs. per hour of the dispersement of carbon black pass through the equipment. 1,255 lbs. per hour of rubber latex (19% by weight solids) is introduced by the latex metering pump. 1,783.35 lbs. per hour of dilute sulphuric acid (1.5% by weight of concentrated sulphuric acid plus water) is introduced by the coagulating pump 54. A suitable anti-oxidant is incorporated, for example—hydroquinone monobenzylether to the extent of 1.25% by weight of dry rubber and this is introduced with the carbon black.

WHAT WE CLAIM IS:—

1. The method of compounding rubber from latex, carbon black and other compounding ingredients in which the operation is carried out continuously by treatment of the rubber latex whilst in the liquid phase as distinguished from plastic mastication, which comprises milling each said other compounding ingredients separately with liquid medium to a colloid size to obtain separate colloidal dispersions of each said other compounding ingredients in the respective media, continuously

metering said separate colloidal dispersions to a common point, continuously metering said carbon black to the same common point to form a master dispersion mixture of compounding ingredients in the combined liquid media, milling said master dispersion mixture to form a master colloidal dispersion, metering said master colloidal dispersion and rubber latex to another common point, mixing said master colloidal dispersion and said rubber latex homogeneously together whereby the particles of said compounding ingredients are impregnated into said rubber latex, all of the above steps beginning with the combining of the compounding ingredients into the master dispersion mixture to the final homogeneous mixture being accomplished continuously and without interruption, thus forming a continuous master mixture.

2. Method according to claim 1, characterized by the further step of separating the rubber and compounding ingredients from the liquid media to form a completely compounded solid rubber mass.

3. Method according to claim 1 or 2, characterized by said carbon black being introduced as a solid and a liquid medium therefor being introduced separately.

4. Method according to claim 1 or 2, characterized by said carbon black being introduced in dispersed form in a liquid medium.

5. Method according to claim 1 or 2, characterized by said carbon black, in powder form, first being compressed, and the resulting carbon black in compressed form being introduced at the first mentioned common point, thereby sealing against back splash of said colloidal dispersions of compounding ingredients into the carbon black feed, and reducing the tendency to form dry core aggregates.

6. Method according to any one of claims 1 to 5, characterized in that an oil is metered directly as such to the first mentioned common point, said oil being thereafter suspended in emulsified form in said master dispersion mixture.

7. The method of compounding rubber substantially as described herein and as shown in the accompanying drawings.

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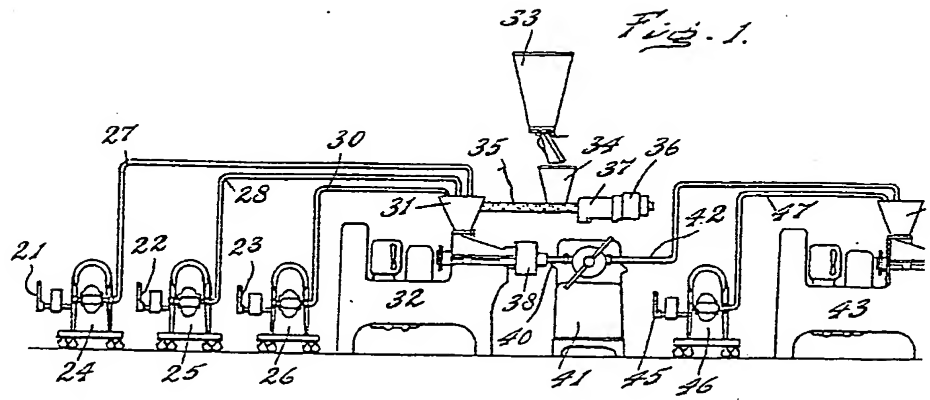
Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1958.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.

dispersion / grinding

elastomer slurry + carbon black (slurry)

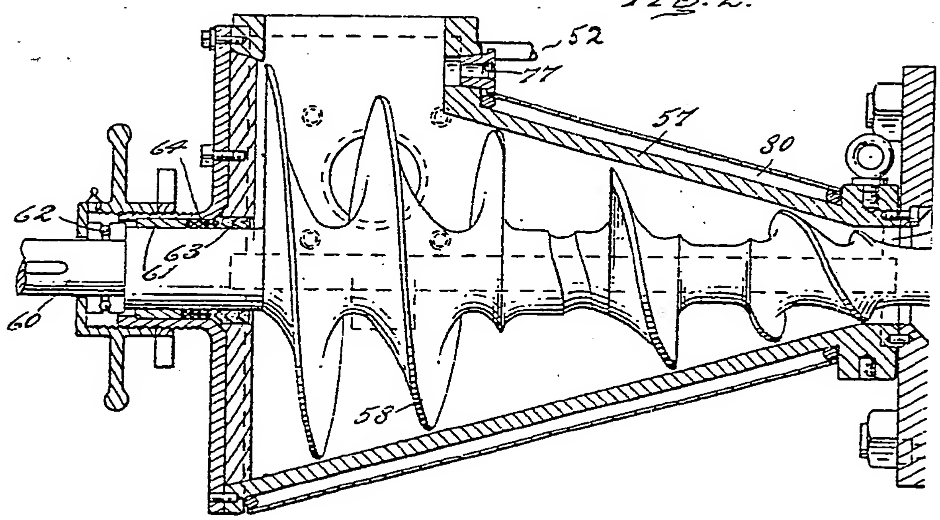
Fig. 1.



coagulation:

+ coagulating agent (e.g. H_2SO_4)

Fig. 2.



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1 SHEET

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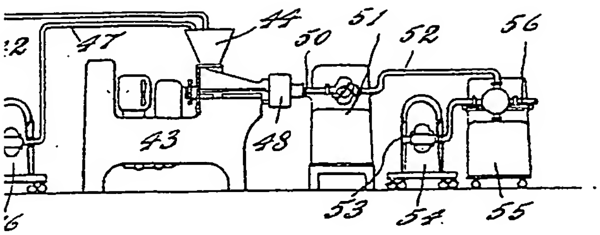

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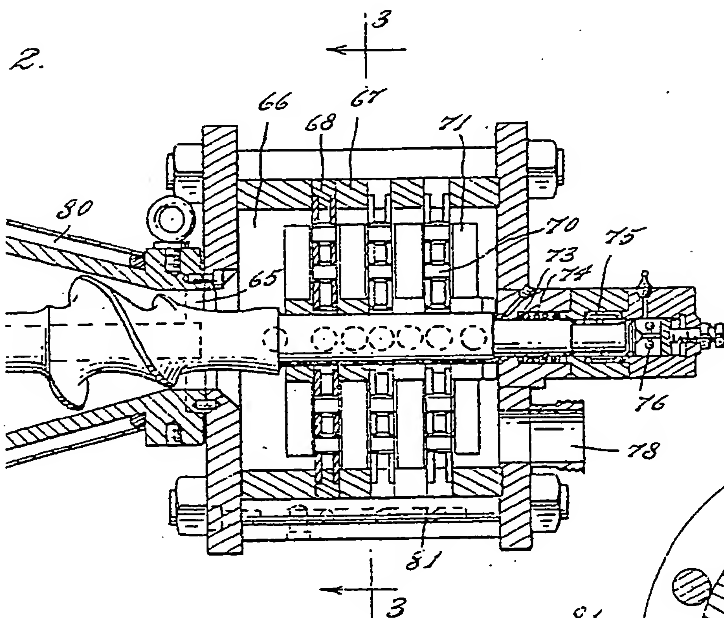

Fig. 3.

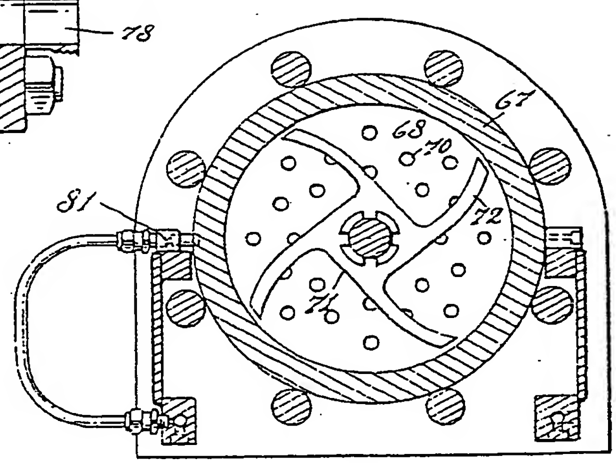

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Fig. 1.

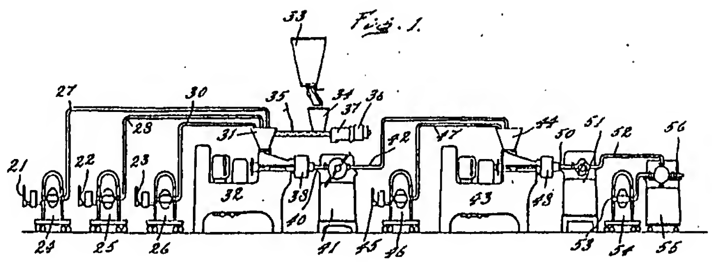

Fig. 2.

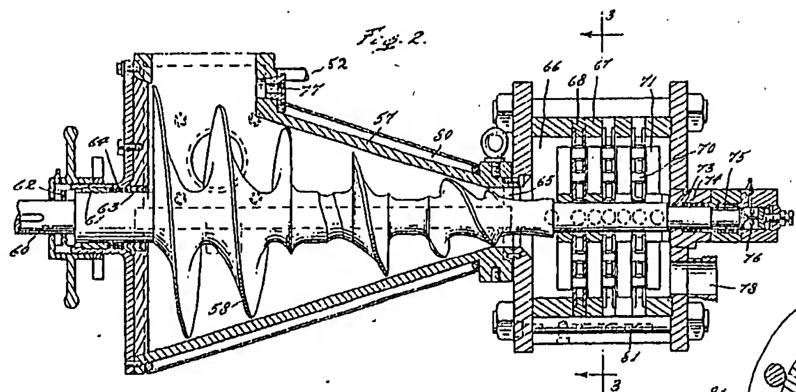

Fig. 3.